UNCLASSIFIED

Registration No. 21143

-Technical Report-

Evaluation of Portable Near Infrared Fuel Analysis Spectrometer

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AUGUST 2010

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Warren, Michigan 48397-5000

UNCLASSIFIED

Report Documentation Page

Form Approved
OMB No. 0704-0188

1. REPORT DATE	2. REPORT TYPE	3. DATES COVERED
10 AUG 2010	**N/A**	**01 JUN 2010 - 10 AUG 2010**

4. TITLE AND SUBTITLE
Evaluation of Portable Near Infrared Fuel Analysis Spectrometer

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
Joel Schmitigal

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
US Army RDECOM-TARDEC 6501 E 11 Mile Rd Warren, MI 48397-5000, USA

8. PERFORMING ORGANIZATION REPORT NUMBER
21143

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
US Army RDECOM-TARDEC 6501 E 11 Mile Rd Warren, MI 48397-5000, USA

10. SPONSOR/MONITOR'S ACRONYM(S)
TACOM/TARDEC

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
21143

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release, distribution unlimited

13. SUPPLEMENTARY NOTES
The views, ipinion, and/or findings contained in this report are those of the authors and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documents., The original document contains color images.

14. ABSTRACT
The objective of this effort was to evaluate the FuelEx portable Near Infrared (NIR) Spectrometer fuel analyzer, by Bruker Optics Inc., developed under guidance from TARDEC under Small Busines Innovative Research contract DAAE07-01-C-L008. The portable spectrometer was evaluted to determine the chemometic effectiveness of the spectrometer's models, providing for an analysis of chemometric model's ability to accurately correspond to known fuel property values as determined by standard ASTM test procedures, evaluate the repeatability of an instrument when measuring the same fuel multiple times and calculate the reproducibility to evaluate the variation of the four instruments being tested.

15. SUBJECT TERMS
Fuel, Diesel, JP-8, Chemometrics, Near Infrared Spectrometry

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			
unclassified	**unclassified**	**unclassified**	**SAR**	**17**	

U.S. Army Tank Automotive Research Development and Engineering Center
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Evaluation of Portable Near Infrared Fuel Analysis Spectrometer

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Force Projection Technology

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1. **Introduction**

The objective of this effort was to evaluate the FuelEx portable Near Infrared (NIR) Spectrometer fuel analyzer, by Bruker Optics Inc., developed under guidance from TARDEC under Small Business Innovative Research contract DAAE07-01-C-L008. The 4 Spectrometers evaluated under this effort were received by The U.S. Army Research Development and Engineering Command's Tank Automotive Research Development and Engineering Center (RDECOM-TARDEC) under contract W56HZV-08-C-L534, an effort that ruggedized the instrument to meet the Army's environmental requirements. The NIR spectrometer is being developed for utilization in the Army's Petroleum Test Kit (1) which will provide fuel handlers with a last quality check of the fuels usability, including fuel type, immediately prior to vehicle or aircraft fueling.

The FuelEX NIR spectrometer utilizes Partial Least Squares (PLS) chemometrics to predict the properties of the fuel and determines the usability of the fuel based on these values as has been detailed in previous reports (2) (3) (4) (5) (6).

2. **Approach**

The NIR fuel analyzer verification was performed to provide an analysis into three instrument factors. Firstly to evaluate the chemometic effectiveness of the spectrometer's models, providing for an analysis of chemometric model's ability to accurately correspond to known fuel property values as determined my standard ASTM International test procedures. Secondly to evaluate the repeatability of an instrument when measuring the same fuel multiple times. Lastly the reproducibility was calculated to evaluate the variation of the four instruments being testing.

The four NIR fuel analyzers were subjected to 32 fuel samples, consisting of Diesel No. 2 (DL-2), JP-8, JP-5, B100 Biodiesel, and gasoline as detailed in Table 1. Each fuel sample was measured 8 times, 4 times each under the Diesel No. 2 and Jet fuel property models, for a total of 1032 sample spectra analyzed. To establish the repeatability of the instrument five jet fuels and five diesel fuels where subjected to 50 additional spectral measurements each, for a total of 500 spectra. After all measurements where taken the data was analyzed to calculate the repeatability of the instruments, and the reproducibility between the instruments for comparison purposes to standard ASTM test procedures.

Sample ID	Fuel Type
737	JP-8
740	JP-8
756	JP-8
758	gasoline
759	Diesel No. 2
761	Diesel No. 2
763	JP-8
766	JP-8
769	Diesel No. 1
880	Diesel No. 2
881	Diesel No. 2
884	Diesel No. 2
890	JP-8
1036	Diesel No. 2
1038	Diesel No. 2
1041	JP-8
1043	Diesel No. 2
1238	JP-5
1304	Diesel No. 2
12770	JP-8
12854	Diesel No. 2
12855	Diesel No. 2
12988	JP-8
12996	JP-8
13014	Diesel No. 2
13119	B100 Biodiesel
13120	Diesel No. 2
13141	JP-8
13201	Diesel No. 2
13301	JP-8
13313	Diesel No. 2
13344	JP-8

Table 1. Test Fuels

3. **Analysis**

3.1. NIR Accuracy

NIR accuracy was analyzed by three methods, 1) fuel specification characterization; 2) NIR property prediction within 1.5 times of the published ASTM reproducibility; 3) Standard Error of Prediction (SEP). Method 1, fuel specification characterization, specifically provides the data output that is required of the NIR spectrometer in the Petroleum Test Kit application, that of providing the Soldier with a last quality check of the fuel; this includes providing a fuel specification characterization and acceptability for use check against published fuel specifications. Comparison of the instruments predicted values against the known ASTM values determined in a laboratory and the accepted reproducibility of the ASTM test methods provides an indication of the NIR method's ability to accurately predict the fuel's properties relative to ASTM accepted values. Method 3 of calculating the Standard Error of Prediction is a standard method for evaluating chemometic models in that it is the standard deviation of all the NIR predicted values from the accepted ASTM values.

3.2. Fuel Specification Categorization

The NIR fuel analyzer was able to accurately identify all 32 of the fuels analyzed, as required by the Petroleum Test Kits requirements documents (1) for each of the 1032 spectra analyzed. The NIR fuel analyzer correctly identified gasoline and biodiesel (B100), as not being an acceptable fuel type, either jet fuel or a DL-2, but was unable to correctly characterize and identify the fuels as there are not chemometric models for these fuels built into the instrument. The only instance where a fuel was incorrectly categorized was when the JP-5 sample was categorized as an acceptable jet fuel but, the samples flashpoint was predicted in the range of 53-57°C, which is below the required 60°C for a JP-5 fuel, the ASTM measured value was 63°C. This result is typical as chemometic modeling of spectroscopic data has not shown a high affinity for accurately predicting flashpoint, the incorrect result could also be due to using a model based on JP-8 fuels which typically have a lower flashpoint than JP-5. The Diesel No. 1 fuel that was analyzed was properly characterized as a Jet Fuel as well as Diesel No. 1 fuel.

3.3. Accuracy to ASTM Values

The NIR fuel analyzer's prediction accuracy to standard ASTM measurements for density (6), cetane index (7), aromatic content (8), distillation point temperatures (9), flashpoint (10), cloud point (11), freeze point (12) and viscosity at -20°C and 40°C (13) was determined to be acceptable for most properties, when utilizing 1.5 times ASTM's published reproducibility (1.5R) as the threshold for acceptability, as detailed in Appendix A. Table 2 details the ASTM repeatability and reproducibility values.

Density and viscosity measurements were found to be outside the 1.5R threshold over 50% of the time, despite those properties having a low Standard Error of Prediction (SEP) due to their ASTM techniques having such low repeatability and reproducibility it is very difficult for chemometic models to provide this level of precision. An average Standard Error or Prediction of 0.005g/mL for density, 0.3cSt for viscosities at -20ºC and 40ºC while not being within the 1.5R threshold, have enough precision for qualifying fuel and allowing for the discrimination between fuel types, and to ensure compatibility for proper engine operation.

Property	Method	fuel type	r, repeatability	R, Reproducibility	Units
Density	ASTM D 1298	all	0.0005	0.0012	g/mL
Cetane Index	ASTM D 4737	all	N/A	N/A	
Aromatics	ASTM D 1319	all	1.4	3.0	%
Distillation*	ASTM D 86				°C
Distil. 10%		Diesel No. 2	3.1-5.3	6.6-10.6	°C
Distil. 90%		Diesel No. 2	2.6-3.6	5.5-7.9	°C
Distil. 10%		JP-8	1.9-3.7	4.3-7.6	°C
Distil. 90%		JP-8	2.1-2.9	4.3-6.3	°C
Flash Point	ASTM D 93	all	1.2-2.3	2.9-5.7	°C
Cloud Point	ASTM D 2500	Diesel No. 2	2.0	4.0	°C
Freeze Point	ASTM D 5972	JP-8	0.54	0.8	°C
Viscosity -20°C	ASTM D 445	JP-8	0.03	0.07-0.09	cSt
Viscosity 40°C	ASTM D 445	Diesel No. 2	.01-.02	.03	cSt

Table 2. Summary of ASTM Repeatability and Reproducibility
*Some ASTM methods provide for a calculated r and R based on the ASTM derived data.
In these cases the test fuel's r and R range are provided.

The flashpoint predictions fell outside of the 1.5R range 32% of the time, this lack of accuracy is not considered important as flashpoint has no impact on the usability of the fuel in diesel and turbine engine applications and the measurement has therefore been determined not to be important for the operational requirements of the Petroleum Test Kit.

3.4. Standard Error of Prediction

Standard Error of Prediction (SEP) is a standard method for evaluating a chemometic models accuracy in that it is the standard deviation of all the NIR predicted values from the accepted ASTM values. The average SEP, for all four instruments utilized in this evaluation, for the samples measured is given in Table 3.

Property		Average JP-8 SEP	Average Diesel SEP
Density	(g/mL)	0.004	0.004
Cetane Index		2.3	0.9
Aromatics	(%)	1.2	3.5
Distil. 10%	(°C)	8.0	12.5
Distil. 90%	(°C)	6.2	6.7
Flash Point	(°C)	6.9	8.8
Cloud Point	(°C)		2.7
Freeze Point	(°C)	3.4	
Viscosity -20°C	(cSt)	0.3	
Viscosity 40°C	(cSt)		0.3

Table 3. Standard Errors of Prediction for evaluated fuels

3.5. NIR Precision

The repeatability and reproducibility of the 4 NIR fuel analyzers and chemometic models compared well to that of the published values of the standard ASTM measurement practices, as shown in tables 4-7. NIR repeatability was calculated using 95% probability and the average standard deviation of measurements performed using the on the same instrument of 50 measurements performed using the same instrument on 5 samples of each fuel type over an 8 hour period of time. The reproducibility was calculated using a 95% probability from the average standard deviation of the measurements for the 32 samples measured 4 times each.

Property		Average NIR Standard Deviation	NIR Repeatability	ASTM Repeatability
Density	(g/mL)	0.0007	0.0018	0.0005
Cetane Index		0.2	0.6	N/A
Aromatics	(%)	0.3	0.7	1.4
Distil. 10%	(°C)	2.1	5.4	1.9-3.7
Distil. 90%	(°C)	0.5	1.4	2.1-2.9
Flash Point	(°C)	1.3	3.2	1.2-2.3
Freeze Point	(°C)	0.48	1.25	0.54
Viscosity -20°C	(cSt)	0.05	0.12	0.03

Table 4. NIR Repeatability of Jet fuel models.
Calculated using 95% probability and average standard deviation of 50 measurements of 5 jet fuel samples measured the same instrument over an 8 hour time period. ASTM values taken from ASTM method documents (www.astm.org).

Property		NIR Standard Deviation	NIR Reproducibility	ASTM Reproducibility
Density	(g/mL)	0.0008	0.0020	0.0005
Cetane Index		0.1	0.4	N/A
Aromatics	(%)	0.4	1.0	1.4
Distil. 10%	(°C)	0.8	2.2	3.1-5.3
Distil. 90%	(°C)	1.7	3.0	2.6-3.6
Flash Point	(°C)	0.7	1.8	1.2-2.3
Cloud Point	(°C)	0.4	1.0	2
Viscosity 40°C	(cSt)	0.05	0.14	.01-.02

Table 5. NIR Repeatability of Diesel No. 2 fuel models.
Calculated using 95% probability and average standard deviation of 50 measurements of 5 jet fuel samples measured the same instrument over an 8 hour time period. ASTM values taken from ASTM method documents (www.astm.org).

Property		NIR Standard Deviation	NIR Reproducibility	ASTM Reproducibility
Density	(g/mL)	0.3652	0.9495	0.0012
Cetane Index		0.3	0.8	N/A
Aromatics	(%)	0.3	0.8	3.0
Distil. 10%	(°C)	2.4	6.2	4.3-7.6
Distil. 90%	(°C)	0.6	1.5	4.3-6.3
Flash Point	(°C)	2.1	5.3	2.9-5.7
Freeze Point	(°C)	1.1	2.8	0.8
Viscosity -20°C	(cSt)	0.60	1.5	0.07-0.09

Table 6. NIR Reproducibility of Jet fuel models.
Calculated using 95% probability and average standard deviation of 15 jet fuel samples measured on 4 different instruments. ASTM values taken from ASTM method documents (www.astm.org).

Property		NIR Standard Deviation	NIR Reproducibility	ASTM Reproducibility
Density	(g/mL)	0.0005	0.0012	0.0012
Cetane Index		0.1	0.2	N/A
Aromatics	(%)	0.3	0.8	3.0
Distil. 10%	(°C)	0.6	1.6	6.6-10.6
Distil. 90%	(°C)	0.8	2.1	5.5-7.9
Flash Point	(°C)	0.8	1.2	2.9-5.7
Cloud Point	(°C)	0.3	0.7	4
Viscosity 40°C	(cSt)	0.05	0.13	0.03

Table 7. NIR Reproducibility of Diesel fuel models.
Calculated using 95% probability and average standard deviation of 15 diesel samples measured on 4 different instruments. ASTM values taken from ASTM method documents (www.astm.org).

4. **Conclusion**

The NIR fuel analyzer was able to correctly identify all 32 of the fuels analyzed, as required by the Petroleum Test Kits requirements documents (1). The NIR fuel analyzer correctly identified gasoline and biodiesel, as not being an acceptable fuel type, either jet fuel or a DL-2, but was unable to identify characterize and identify the fuels as there are not chemometric models for these fuels built into the instrument. The only instance where a fuel was incorrectly categorized was when the JP-5 sample was categorized as a jet fuel but not as JP-5 as its flashpoint was predicted to be between 53-57°C which is below the required 60°C, the ASTM measured value for this sample was 63°C.

The NIR fuel analyzer was able to provide agreement with standard ASTM measurements, with the exception of density and viscosity measurements which have such low repeatability and reproducibility that it is very difficult for chemometic models to provide this level of precision.

The NIR fuel analyzer chemometic models had repeatability on par with that of utilizing standard ASTM measurement practices. The reproducibility between the four NIR fuel analyzers tested was observed be less than when utilizing standard ASTM measurement practices for most properties.

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Appendix A

	Aromatics	Density	Distil. 10%	Distil. 90%	Flash Point	Freeze Point	Viscosity -20°C
13344	Yes	Yes	Yes	Yes	Yes	Yes	No
13301	Yes	Yes	Yes	No	Yes	Yes	No
13141		No	Yes	Yes	Yes	No	
12996	Yes	No	Yes	Yes	No	No	No
12988	Yes	No	Yes	Yes	No	No	No
1238		No			No		No
1041	Yes	Yes	Yes	Yes	Yes		Yes
890	Yes	No	No	Yes	Yes	No	
769		No			yes		
740	Yes	No	Yes	Yes	Yes	Yes	

Table A1. NIR jet fuel property prediction overlap ASTM-value ± 1.5R (instrument 2004)

	Aromatics	Density	Distil. 10%	Distil. 90%	Flash Point	Freeze Point	Viscosity -20°C
13344	Yes	No	Yes	Yes	Yes	No	Yes
13301	Yes	No	Yes	No	Yes	Yes	No
13141		Yes	Yes	Yes	Yes	No	
12996	Yes	No	No	Yes	No	No	No
12988	Yes	No	No	Yes	No	No	No
1238		Yes			Yes		Yes
1041	Yes	Yes	Yes	Yes	Yes		No
890	Yes	Yes	Yes	Yes	Yes	Yes	
769		No			yes		
740	No	Yes	Yes	Yes	No	No	

Table A2. NIR jet fuel property prediction overlap ASTM-value ± 1.5R (instrument 2005)

	Aromatics	Density	Distil. 10%	Distil. 90%	Flash Point	Freeze Point	Viscosity -20°C
13344	Yes	No	Yes	No	Yes	No	Yes
13301	Yes	No	Yes	No	Yes	Yes	No
13141		Yes	Yes	Yes	Yes	No	
12996	Yes	No	No	Yes	No	No	No
12988	Yes	No	Yes	Yes	No	No	No
1238		Yes			Yes		Yes
1041	Yes	No	Yes	Yes	Yes		No
890	Yes	Yes	Yes	Yes	Yes	No	
769		No			Yes		
740	Yes	Yes	Yes	Yes	Yes	Yes	

Table A3. NIR jet fuel property prediction overlap ASTM-value ± 1.5R (instrument 2006)

	Aromatics	Density	Distil. 10%	Distil. 90%	Flash Point	Freeze Point	Viscosity -20°C
13344	Yes	Yes	Yes	No	Yes	No	No
13301	Yes	No	Yes	No	Yes	Yes	No
13141		Yes	Yes	Yes	Yes	No	
12996	Yes	No	No	Yes	No	No	No
12988	Yes	No	Yes	Yes	No	No	No
1238		Yes			Yes		No
1041	Yes	Yes	Yes	Yes	Yes		No
890	Yes	No	No	Yes	Yes	No	
769		No			Yes		
740	Yes	Yes	Yes	Yes	Yes	Yes	

Table A4. NIR jet fuel property prediction overlap ASTM-value ± 1.5R (instrument 2010)

	Aromatics	Cloud Point	Density	Distil. 10%	Distil. 90%	Flash Point	Viscosity +40°C
13313	Yes	Yes	No	Yes	Yes	No	
13201	Yes	Yes	No	Yes	No	No	No
13120	Yes	Yes	No	Yes	Yes	No	No
13014	Yes	Yes	No	No	Yes	No	No
12855	Yes	Yes	Yes	No	Yes	Yes	No
12854	Yes		No	No	Yes	Yes	No
1038	No		No	Yes	Yes	Yes	Yes
1036		Yes	No	Yes	Yes	Yes	No
881	Yes		No			Yes	No
761	Yes		No	Yes	Yes	Yes	Yes
759	Yes		Yes	Yes	Yes	Yes	No

Table A5. NIR diesel fuel property prediction overlap ASTM-value ± 1.5R (instrument 2004)

	Aromatics	Cloud Point	Density	Distil. 10%	Distil. 90%	Flash Point	Viscosity +40°C
13313	Yes	Yes	No	Yes	Yes	No	
13201	Yes	Yes	Yes	Yes	No	No	No
13120	Yes	Yes	Yes	Yes	Yes	No	No
13014	Yes	Yes	No	No	Yes	No	No
12855	Yes	Yes	Yes	No	Yes	Yes	No
12854	Yes		No	Yes	Yes	Yes	No
1038	No		No	Yes	Yes	Yes	No
1036		Yes	Yes	Yes	Yes	Yes	No
881	Yes		Yes			Yes	No
761	Yes		Yes	Yes	Yes	Yes	No
759	Yes		No	Yes	Yes	Yes	No

Table A6. NIR diesel fuel property prediction overlap ASTM-value ± 1.5R (instrument 2005)

	Aromatics	Cloud Point	Density	Distil. 10%	Distil. 90%	Flash Point	Viscosity +40°C
13313	No	Yes	Yes	Yes	Yes	No	
13201	No	Yes	No	Yes	No	No	No
13120	No	Yes	No	Yes	Yes	No	No
13014	No	Yes	No	No	Yes	No	No
12855	No	Yes	No	No	Yes	Yes	No
12854	Yes		Yes	Yes	Yes	Yes	No
1038	Yes		No	Yes	Yes	Yes	No
1036		Yes	No	Yes	Yes	Yes	No
881	Yes		No			Yes	No
761	Yes		No	Yes	Yes	Yes	No
759	Yes		No	Yes	No	Yes	No

Table A7. NIR diesel fuel property prediction overlap ASTM-value ± 1.5R (instrument 2006)

	Aromatics	Cloud Point	Density	Distil. 10%	Distil. 90%	Flash Point	Viscosity +40°C
13313	Yes	Yes	No	Yes	Yes	Yes	
13201	Yes	Yes	Yes	Yes	No	No	No
13120	No	Yes	Yes	Yes	Yes	No	No
13014	Yes	Yes	No	No	Yes	No	No
12855	Yes	Yes	Yes	No	Yes	Yes	No
12854	Yes		No	Yes	Yes	Yes	No
1038	Yes		Yes	Yes	Yes	Yes	No
1036		Yes	Yes	Yes	Yes	Yes	Yes
881	Yes		Yes			Yes	No
761	Yes		No	Yes	Yes	Yes	No
759	Yes		No	No	Yes	No	No

Table A8. NIR diesel fuel property prediction overlap ASTM-value ± 1.5R (instrument 2010)

List of Symbols, Abbreviations, and Acronyms

°C	degree(s) Celsius
cSt	centistokes
Distil.	Temperature at distillation boiling point
DL-1	Diesel Fuel No.1
DL-2	Diesel Fuel No.2
g	Grams
JP-5	Jet Propellant 5
JP-8	Jet Propellant 8
mL	Milliliter
NIR	Near Infrared
PLS	Partial Least Squares
r	Repeatability
R	Reproducibility
RDECOM	Research Development and Engineering Command
SEP	Standard Error of Prediction
TARDEC	Tank Automotive Research Development and Engineering Center